



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification³ : C08L 63/02	A1	(11) International Publication Number: WO 83/ 01454 (43) International Publication Date: 28 April 1983 (28.04.83)
(21) International Application Number: PCT/US82/00821 (22) International Filing Date: 14 June 1982 (14.06.82) (31) Priority Application Number: 313,625 (32) Priority Date: 21 October 1981 (21.10.81) (33) Priority Country: US (71)(72) Applicant and Inventor: SMITH, Charles, E. [US/ US]; 2307 Raintree, Frankfort, KY 40602 (US). (74) Agent: STEUTERMANN, Edward, M.; 1332 So. Sec- ond St., Louisville, KY 40208 (US). (81) Designated States: AT (European patent), AU, BR, CF (OAPI patent), CG (OAPI patent), CH (European pa- tent), CM (OAPI patent), DE (European patent), DK, FR (European patent), GA (OAPI patent), GB (Euro- pean patent), JP, LU (European patent), NL (Euro- pean patent), NO, SE (European patent), SN (OAPI patent), SU, TD (OAPI patent), TG (OAPI patent).		Published <i>With international search report.</i>
(54) Title: A METHOD AND COMPOSITION FOR SUBSTRATE COATING (57) Abstract A method and composition for coating a selected substrate including mixing a first liquid solution of first solvent and an epoxy resin, with a second solution including selected proportions of epoxy resin curing agent reactive with the epoxy resin of the first solution, second solvent, selected proportions of a selected wetting agent soluble in both water and in generally water insoluble liquid and a selected proportion of water, and applying the mixed first and second solutions to a selected substrate.		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	LI	Liechtenstein
AU	Australia	LK	Sri Lanka
BE	Belgium	LU	Luxembourg
BR	Brazil	MC	Monaco
CF	Central African Republic	MG	Madagascar
CG	Congo	MR	Mauritania
CH	Switzerland	MW	Malawi
CM	Cameroon	NL	Netherlands
DE	Germany, Federal Republic of	NO	Norway
DK	Denmark	RO	Romania
FI	Finland	SE	Sweden
FR	France	SN	Senegal
GA	Gabon	SU	Soviet Union
GB	United Kingdom	TD	Chad
HU	Hungary	TG	Togo
JP	Japan	US	United States of America
KP	Democratic People's Republic of Korea		

-1-

A METHOD AND COMPOSITION FOR SUBSTRATE COATING

DESCRIPTION

TECHNICAL FIELD

My invention relates to epoxy coating particularly useful in providing a coating for selected substrates where the coating provides improved adherence to the substrate by improved penetration of the coating into the substrate.

In general, epoxy compounds are composed of monomers or prepolymers that further react with curing agents to yield high performance thermosetting plastics. Epoxy compounds have gained wide acceptance as protecting coatings and in structural applications because of their exceptional combination of properties such as toughness, adhesion, chemical resistance, and superior electrical properties. Epoxy resins are Generally characterized by the presence of a three member cyclic ether group commonly referred to as an epoxy group, 1,2-epoxide, or oxirane. The most widely used epoxy resins are diglycidyl ethers of bisphenol A



-2-

derived from bisphenol A and epichlorhydrin
as typically shown in United States Patent
No. 3,324,483 and U.S. Patent No.
2,456,408-Castan.

5 Epoxy resins are most frequently cured
with anhydrides aliphatic amines or
polyamides.

BACKGROUND ART

 A variety of agents have been
10 described for converting liquid epoxy resins
to a cured state, which is necessary for the
development of the inherent properties of
the resins. The curing agents or hardners
are categorized generally as catalytic or
15 co-reactive. The functional groups of the
resins are terminal epoxy groups together
with pendant hydroxyl per repeat unit of the
polymer chain.

 Co-reactive curing agents are
20 generally employed in stoichiometric
quantities with the epoxy resin. The



-3-

important classes include polyamines, polyaminoamides, polyphenols, polymeric thiols, polycarboxylic acid and anhydrides.

Most systems utilized in current practice are solvent based, that is the resin, or the curing agent, or both, are dissolved in selected solvents for ease in application. In general the solvents and the systems are generally water free.

Recently, water borne systems have been developed for replacement of solvent based systems. In general the water borne systems utilize either liquid or solid epoxy resins which have been modified to allow use with water. They are usually in the form of emulsions, suspensions, dispersions or water dilutable resins which can be heat or room temperature cured.

DISCLOSURE OF INVENTION

The present invention provides a new and useful composition and method which provides unexpectedly improved penetration



-4-

of the resin into pores of the substrate for better protection and greatly improved bonding between the substrate and the coating. Various substrates can be utilized
5 where the composition of the coating can be varied to accomodate the character of the substrate. The substrates can be, for example, emulsified asphalt, cement, masonry materials or even metals. Further, many
10 different types of fillers and extenders such as rock, sand, glass, carborundum, alumina, quartz, common slag, marble, grit, or granite can be incorporated to provide improved wear resistance. The resulting
15 product is water proof, resistant to acid and alkali solutions and semi-flexible, yet very hard and strong and adheres tightly to the substrate, it is believed, because of the wetting agent which is a part of the
20 invention and promotes resin penetration of the substrate.

In application it has been found that



-5-

the compositions provided by the present invention provide enhanced protection to bridge decks to prevent water and salt penetration and as a base for lead bearing coatings to provide radiation shielding for selected form of radiation. Further compositions within the scope of the present invention provide improved bonding with greatly reduced substrate surface preparation, for example by sandblasting or degreasing.

Additionally, materials within the scope of the present invention can be modified to provide fire and thermal resistance and the material can be provided on various substrates such as metals, cloth, paper, fiberglass, burlap and various plastics.

Also the material is quick setting so that the substrate can be returned to use shortly after application of the material.



-6-

More particularly the present invention provides an improved method and composition for coating a selected substrate including mixing a first liquid solution of first solvent and an epoxy resin, with a second solution included selected proportions of epoxy resin curing agent reactive with the epoxy resin of the first solution, second solvent, selected proportions of a selected wetting agent soluble in both water and in generally water insoluble liquid and a selected proportion of water, and applying the mixed first and second solutions to a selected substrate.

BEST MODE FOR CARRYING OUT THE INVENTION

Examples within the scope of the present invention are discussed hereinafter and it will be understood that the examples are by way of illustration only and that various other compositions also within the scope of the present invention will occur to those skilled in the art upon reading the disclosure set forth hereinafter.



-7-

In accordance with the present invention it has been unexpectedly found that the use of a wetting agent in combination with an epoxy resin-curing agent system unexpectedly provides a base coat for application to a substrate which provides an unusually strong and effective substrate bond where the first coat can be then utilized as a base for application for subsequent coats of epoxy or other materials or where the material can, for example, be used as a grout or filler for example for concrete.

Further, in accordance with the present invention it has been found that in the epoxy resin-curing agents systems the presence of solvents in the curing agent and epoxy resin facilitate the development of properties of the desired properties in the overall composition. Additionally, in some instances application of more than one coat of the base material utilizing the wetting



-8-

agent as described hereinafter has been found useful where the composition of the material used in the first and second applications may be varied.

5 In one example in accordance with the present invention a system was compounded including a solvent bond epoxy resin (Epi-rez™ 2036 product of the Celanese Plastics and Specialties Company) having a
10 75% by weight resin as solid in solution with metholisobutylketone solvent at approximately 16.6% by weight and xylene solvent at approximately 8.9% by weight. The curing agent used in stoichiometric
15 amount included Epi-Cure™ CT-60-8534 by Celanese Plastics and Specialties Company as aliphatic amine in solution of solvents at concentrations of 8% by weight toluene, 8%
20 by weight ethylene glycol monoethylether and 24% by weight ethylene glycol monobutyl ether.

An example wetting composition within



-9-

the scope of the present invention was formulated in accordance with the following formulation by weight:

	Water	10%
5	Methylethyl Ketone	10%
	Dimethyl Sulfoxide	64%
	Isopropyl Alcohol	16%

The Methylethyl Ketone, Dimethyl Sulfoxide, and Isopropyl Alcohol act as a wetting agent to allow the water in the total mixture to be taken into the solution first with the curing agent and finally with the resin base. Other wetting agents can be utilized within the scope of the present invention and can include detergent compositions.

An example formulation was then compounded wherein the wetting agent included at a concentration of 5% of the final composition, was mixed with the Epi-Cure™ CT-60-8534 which was then mixed with the resin solution so in the final mix

-10-

the curing agent was 47.5% by weight of the final composition the wetting agent was 5% and the balance (47.5% by weight) of the composition was the Epi-Res™ 2036.

5 The material was then applied to a surface to be coated as compounded and allowed to harden. In some applications further dilution of the penetrating composition has been found advantageous. It
10 has been found that active compositions can be prepared wherein the aforementioned composition including resin, curing agent and wetting agent is diluted in water at a ratio of three parts water to one part of
15 the composition.

 While various compositions have been
4 found to be satisfactory it has been found that so long as sufficient wetting agent is present to provide a complete solution of
20 the water in the epoxy system, the system is effective. In this regard it is believed that the wetting agent need only be a material which is soluble in polar solvent



-11-

composition including resin, curing agent and wetting agent is diluted in water at a ratio of three parts water to one part of the composition.

5 While various compositions have been found to be satisfactory it has been found that so long as sufficient wetting agent is present to provide a complete solution of the water in the epoxy system, the system is
10 effective. In this regard it is believed that the wetting agent need only be a material which is soluble in polar solvent such as water and non polar solvents such as the xylene, or other solvents normally found
15 in resin-curing agent systems.

 In application, the composition previously described is diluted with water as previously described. A first coat can be applied at the rate of, for example, 300
20 square feet per gallon. The coating is allowed to dry and a second coat, which is somewhat less diluted (for example where the



-12-

resin, curing agent, wetting agent, is diluted with two parts water) is applied over the first coat and allowed to dry. As a third coat the solution utilizing the wetting agent with:

Water	10%
Methylethyl Ketone	10%
Dimethyl Sulfoxide	64%
Isopropyl Alcohol	16%

is utilized and mixed with curing agent and epoxy resins so the wetting agent is 5% of the total mixture. Thus the curing agent is 47 1/2% of the final mixture and the epoxy resin is 47 1/2% of the final mixture. The third coat can be mixed with abrasive or wear resistant material when a protected surface is desirable or when surface roughness is needed.

In this regard it has been further found that by utilization of one or two coats of the material including the wetting agent as a precoat on concrete, steel, or other material, and the subsequent addition



-13-

of epoxy material either with or without the wetting composition, but mixed with up to 95% lead powder and applied to the precoated surface, an extremely efficient radiation shield is provided. A test was performed utilizing epoxy resin mixed with lead powder where the epoxy resin was approximately 2 to 3% of the mixture, the lead being the balance, as a radiation shield and the composition then tested with reference to equal thickness of steel and aluminum. The tests were performed utilizing samples of quarter inch thickness increments and varying the exposure time and the X-ray intensity in accordance with the data shown in Table I. In Table I the relative X-ray intensity is reflected by the combination of the voltage and current to the X-ray. The thickness of the materials exposed for the times indicated is also shown and the relative transmission of the various materials under each of the conditions is shown.

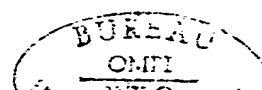
-14-

TABLE I
X-RAY TRANSMISSION DATA

THICKNESS OF BARRIER INCHES	X-RAY KILOVOLT	MILLIAMP	EXPOSURE TIME-MIN
0.25	140	20.0	3
0.5	200	15.0	3
0.75	220	12.0	4
1.0	280	10.0	3
1.25	300	10.0	5
1.5	300	10.0	10
1.75	300	10.0	15

BARRIER & RELATIVE
TRANSMISSION-%

Al	Fe	Epoxy Lead
62.5	25	0
100	50	0
100	50	0
100	87.5	6
100	87.5	6
100	100	12.5
100	100	2.0



-15-

It will be seen that the epoxy-lead composition is highly effective in reducing X-ray transmission and that materials of this type which can be bonded to selected
5 substrates using compositions within the scope of the present invention would be particularly effective as radiation shielding.

It has further been found that fire
10 retardant materials can be easily compounded with materials within the scope of the present invention to substantially provide fire retardant and in some cases fire protective shielding.

15 It has further been found that compositions within the scope of the present invention can be admixed with substrate material, for example concrete to provide improved characteristics, particularly
20 improved resistance to element penetration, reduction of reinforcing steel corrosion and reduction of spalling due to ice and salt.



-16-

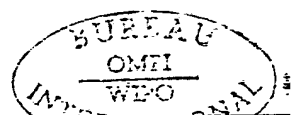
It will be recognized that the foregoing description of the present invention is not by way of limitation but that various other formulations, compositions, and applications also within the scope of the present invention will occur to those skilled in the art upon reading the disclosure set forth hereinbefore.



-17-

CLAIMS

- 1 A coating composition for coating a
selected substrate including a mixture
of: a) from 30 to 75% by weight of a
liquid epoxy resin including active
5 epoxy groups; b) a stoichiometric
quantity of selected liquid curing agent
for said liquid epoxy resin including at
least 2.5% by weight of water and a
selected proportion of a selected
10 wetting agent soluble in polar and
nonpolar liquids in quantities
sufficient to effect a solution of said
water, wetting agent, curing agent and
liquid epoxy resin.
- 15 2 The invention of Claim 1 wherein said
liquid epoxy resin includes at least one
nonpolar solvent.
- 3 The invention of Claim 1 wherein said
curing agent is co-reactive with said
20 epoxy groups.
- 4 The invention of Claim 1 wherein said
curing agent promotes catalytic



-18-

polymerization of said epoxy groups.

5 The invention of Claim 1 wherein said
curing agent includes nonpolar solvent.

6 The invention of Claim 1 wherein said
5 wetting agent includes alcohol.

7 The invention of Claim 6 wherein said
alcohol includes propylalcohol.

8 The invention of Claim 1 wherein said
wetting agent includes

10 dimethylsulfoxide.

9 The invention of Claim 1 wherein said
wetting agent includes ketone groups.

10 The invention of Claim 9 wherein said
ketone group is methylethylketone.

15 11 The invention of Claim 1 wherein said
wetting agent is from 3 to 6% of the
weight of said coating composition.

12 The invention of Claim 1 including a
second composition provided by diluting
20 said coating composition with water.

13 The invention of Claim 12 wherein said
coating composition is diluted at least
in equal parts with water.



-19-

- 14 The invention of Claim 1 wherein said
composition is admixed with a calcium
aluminate calcium silicate composition
which hydrates in the presence of water
5 to form a hardened substance.



INTERNATIONAL SEARCH REPORT

International Application No

PCT/US82/00821

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ³		
According to International Patent Classification (IPC) or to both National Classification and IPC		
INT. CL. ³ C08L 63/02		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁴		
Classification System	Classification Symbols	
U.S.	523/401, 402, 414, 420; 528/121	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁵		
CHEMICAL ABSTRACTS: - TO DATE		
III. DOCUMENTS CONSIDERED TO BE RELEVANT ¹⁴		
Category *	Citation of Document, ¹⁶ with indication, where appropriate, of the relevant passages ¹⁷	Relevant to Claim No. ¹⁸
A	US, A, 4,143,188 PUBLISHED 6 MARCH 1979; COLUMN 3, LINES 36-48; COLUMN 4, LINES 1-66, HUBER-NUESCH	1-14
A	GB, A, 972,801 PUBLISHED 14 OCTOBER 1964; PAGE 1, COLUMN 1, LINES 60-67; PAGE 2 COLUMN 1, LINES 59-65, PAGE 3, COLUMN 1, LINES 30-60, RAYBESTOS- MANHATTAN, INC.	1-14
<p>* Special categories of cited documents: ¹⁵</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&" document member of the same patent family</p>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search ²	Date of Mailing of this International Search Report ²	
28 OCTOBER 1982	21 OCT 1982	
International Searching Authority ¹	Signature of Authorized Officer ²⁰	
ISA/US	THEODORE PERTILLA	